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**Ateeq, M, Shaw, A, Garrett, R and Dickson, P (2016) Feasibility study on using microwave sensing technique to analyse silver-based products. Journal of Electromagnetic Waves and Applications. ISSN 1569-3937**

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# Feasibility study on using Microwave sensing technique to analyse Silver based products

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## Abstract

This paper reports on the feasibility of using a novel and robust microwave sensing technology to detect and analyse various Silver based products such as Silver Nitrate and Silver Oxide. The focus of the investigation is to differentiate between the two products, identify the contamination and change in the sample size. A microwave sensor designed previously in house has been utilised to carry out this initial study to analyse the capability of microwave sensing technique to carry out the analysis. The change in the microwave spectra are used as an indicator of the difference in the silver products and any contamination they may have. The results and their detailed repeatability confirm the viability of using microwave sensing technique as a potential method to analyse various silver products. The curves obtained from the material response to microwaves are distinguishable and can be related to the materials' properties. The study suggests a design and development of a bespoke unit as a dedicated analysis tool and to address any anomalies arising from the current feasibility. This will have a huge industrial benefit in terms of cost reduction and time associated with the industrial analysis.

*Keywords: Microwave sensing; non-destructive analysis; novel microwave analysis; Cavity resonator; silver products; silver products characterisation; industrial analysis.*

## **1. Scope and application of the study**

A feasibility study was carried out as a proof of concept to initially investigate the potential of using a novel microwave based sensing technology to measure and detect silver products of various types, detect any contamination they may have and particle size/particle size distribution in real time. This work was carried out in collaboration with AmesGoldsmith UK Ltd addressing the problem around characterising both the silver nitrate and silver oxide in terms of various properties. Assessing these two products are the most important for Amesgoldsmith UK Ltd in terms of quality assurance and testing. Current practice at the premises of the industrial partner follows conventional mechanical and chemical methods of analysis and testing to study the type of silver products, contamination, particle size, particle size distribution, etc. These methods are time consuming, laborious to carry out and are often inefficient, inaccurate and inconsistent. To overcome these drawbacks, to improve the quality control and assessment of the material manufactured, and to improve the operational efficiency of the organisation, there was a need to come up with more efficient, robust and cost effective testing and analysis method. This feasibility study will provide the industrial partner involved in the work with sufficient information on the potential of introducing a new, innovative and novel method of measurement, analysis and detection. This investigation will also provide a ground for further research work to explore the possibility of carrying out detailed characterisation of the silver products through the use of microwave sensing technique as a real time alternative to conventional methods. Although this work presents a proof of concept, the ultimate aim in the future would be to design and develop a microwave sensing unit of a suitable dimension, size and shape for the industrial environment.

## **2. Background**

Microwave based sensing is a relatively new and rapidly developing technology [1, 2]. It offers a great potential as a developing technology due to its accuracy, low cost of measurements both in static and continuous measurements as well as its capability to analyse samples in small sizes [3, 4]. It is an instantaneous and robust technique, non-invasive in nature, the sensor equipment operates at substantially low power and completely non-ionising in nature, i.e. operating at 0 dBm or 0.001 watt (1 mW). Despite the amount of power utilised it has fairly good penetration depth. It uses Electromagnetic (EM) waves in the microwave band of the spectrum (300 MHz to 300 GHz). It is an efficient technique and largely used for the

characterisation of materials because it can easily propagate through low-loss substances such as plastics, glass, ceramic, etc. [1, 2, 5, 6]. It is a relatively straight forward technique and the instrumentation for measurements can be setup in minutes with the availability of measurement results in seconds providing real-time data [6, 7]. In addition to the advantages above there are certain disadvantages however including a higher degree of specialisation and simultaneous existence of multiple variables such as temperature, density, moisture, structure, etc. affecting the microwave measurements [8].

### **2.1. Microwave theory and applications**

The Microwaves can be used to monitor changes in the permittivity of the material determined by the molecular structure of it. Any change in the molecular structure affects its permittivity properties and is reflected in the microwave spectrum obtained due to the interaction of waves to the material [2, 6, 7, 9]. Permittivity is simply a measurement of the response of a dielectric medium to the applied microwaves in the form of change in its electric field. It is dependent on the material's ability to polarise in response to the applied field. The permittivity  $\varepsilon_r$  of the material is defined in equation (1) [6].

$$\varepsilon_r = \varepsilon' + j\varepsilon'' \quad (1)$$

In equation (1) above,  $\varepsilon'$  represents the energy stored by a material and  $\varepsilon''$  represents any losses of energy [6].

Due to the multi-parameter nature of the microwave analysis technique, the interaction of microwaves with the material provides unique spectrum signatures resulting in the change in the material properties, i.e. its permittivity. This could be due to change in the frequency, attenuation or reflection of the signal measured in the form of scattering parameters also referred to as S-parameters. S-parameters are simply the transmitted  $S_{21}$  and reflected  $S_{11}$  microwave powers. By considering how these parameters change at discrete frequency intervals, the change can be linked to the material type, its composition, concentrations of the constituents, size/size distribution, etc. in the sample [6, 9].

Microwave sensing technique although in its infancy has been implemented in various industrial applications including glucose concentration monitoring [10], water industry including multiphase flow monitoring [11], characterisation of construction materials [12, 13],

food industry [14], water level measurements, material moisture contents, healthcare industry, etc. [9].

### **3. Microwave sensing experimental setup**

The feasibility study utilised cavity based microwave sensors (cavity resonators) to study the properties of silver based products including silver oxide and silver nitrate. Resonance occurs in the cavity resonators when the electric and magnetic fields form a standing wave. A number of electromagnetic wave modes can occur in the cavity and each of these modes have its own resonant peak. Each of the resonant peaks generated by various modes has a quality factor  $Q$ . A high value of  $Q$  represents a sharp peak that may be easily analysed. However, the change in the resonant peak may also be useful to analyse the properties of a material under test [11].

To satisfy the proof of concept criteria two existing resonant cavity sensors were utilised designed at the Radio Frequency & Microwave (RFM) Group of Liverpool John Moores University (LJMU). The reason to utilise two different resonant cavity sensors in the feasibility study was not only to show the effectiveness of the microwave sensors as a potential technology but also to see how various parameters such as the shape and the size can influence resonant frequencies (which are dependent on the dimensions) as well as quality of the results. The dimensions of the cavity has an influence on the modes generated (resonant peaks) and the response frequencies obtained. The final design for the industrial unit can be achieved through a modelling tool such as COMSOL or HFSS taking into consideration the dimensions as well as the types of the material tested, along with the results obtained from this study. For the sake of this study these sensors would be referred to as microwave sensor 1 and microwave sensor 2. The microwave sensor 1 was a pan cake type cavity resonator whereas the microwave sensor 2 was a rectangular type cavity resonator. At the feasibility stage of investigation the focus was on a few parameters such as differentiating between silver oxide and silver nitrate, determination of contamination in the sample as well as sample size/sample size distribution. Although the two used sensors provided measurement capability in different frequency range (based on their size and shape), the overall aim was to show the effectiveness of the sensing technique. The cavity resonator can be classed as a black box where the microwaves are inserted from the input port interacting with the material and leaving through the output port. The interaction of these microwaves with the

material under test helps in characterising it. The aluminium cavities and experimental setup of the sensing system used in the study is shown in Figures 1 and 2. Both the sensors in Figures 1 and 2 have a sample holder in the middle of the cavity, the microwave source, the Vector network analyser (VNA), cables and connectors.

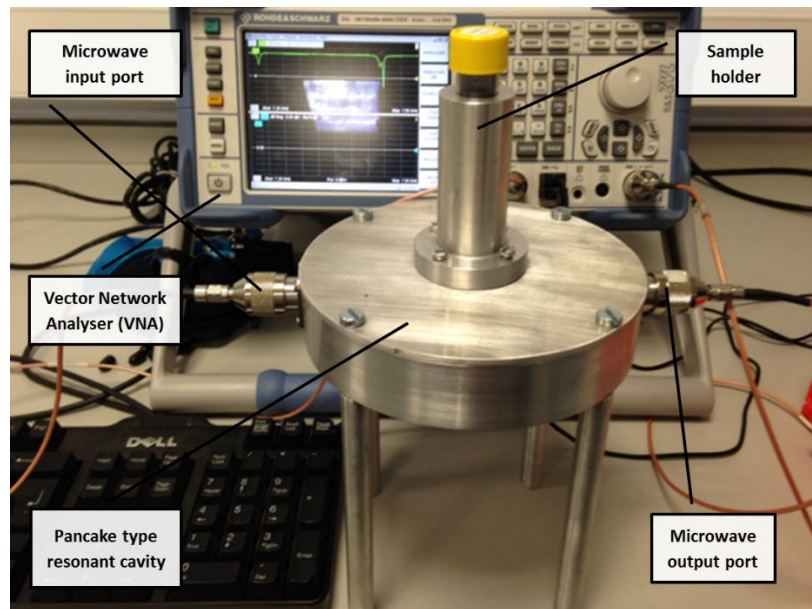


Figure 1: Experimental setup utilising the pancake type microwave sensor 1, cables, connectors and Vector Network Analyser

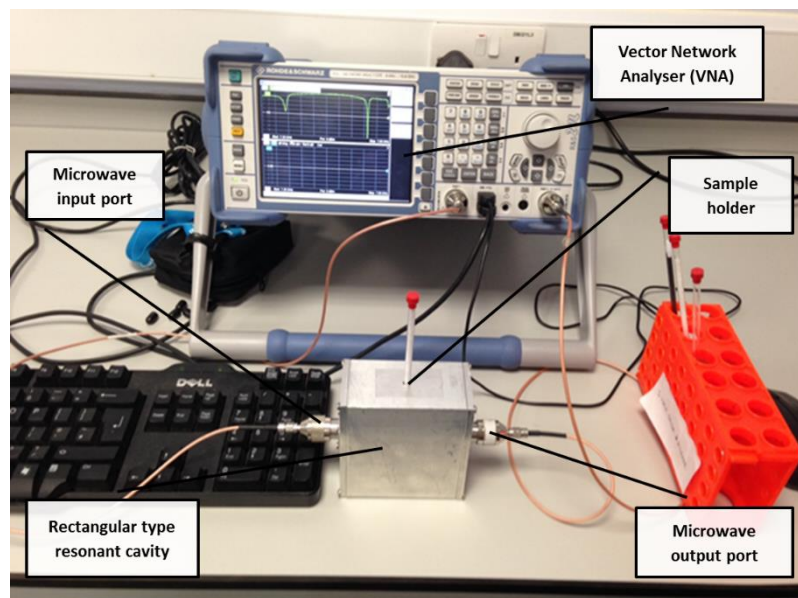


Figure 2: Experimental setup utilising the rectangular type microwave sensor 2, cables, connectors and Vector Network Analyser.

#### 4. Samples measured and preparation

The Silver samples analysed were provided by AmesGoldsmith UK Ltd. They were prepared and tested in two different types of sample tubes. A 15 ml polypropylene sample tube suitable for the analysis in the microwave sensor 1 (Figure 1) and a glass NMR tube suitable for testing in the microwave sensor 2 (Figure 2). All the samples tested along with the types of sample tubes used are shown in Figure 3 (a) & (b).

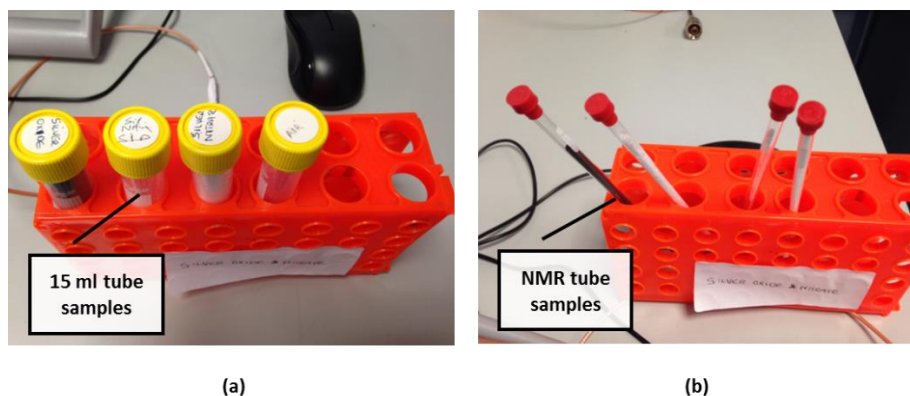


Figure 3: (a) Samples in 15 ml tubes tested in microwave sensor 1 (b) Samples in 15 ml tubes tested in microwave sensor 2

The samples consisted of two types of silver products, i.e. Silver oxide and Silver Nitrate powders. Testing only two types of silver products was in response to the Amesgoldsmith UK Ltd requirements. These two products are the most important for them to be analysed and tested in the initial stage to make their post-production analysis more robust. However, the technique can be expanded to include other silver products in the future to meet the silver products industry demands. Each sample had a particle size range uniformly distributed across the sample tube. The type of samples tested are listed in Table 1 along with their description and type. Both the silver oxide and nitrate were analysed individually. Also, to determine the capability of microwave sensing technique to detect any contamination or foreign material, silver nitrate was mixed with a polymer (polyethylene) as 50% source of contamination as well as to study the change in size distribution. To further study the contamination properties other material might be introduced in future studies to elaborate on the effectiveness of using microwave sensing technique to determine the contamination/foreign material and percentage of it.

Table 1: Silver based samples analysed and tested using microwave sensing technique

Sample #	Sample Abbreviation	Description and Sample Size (approx.) (microns)
<b>Samples tested in pancake type microwave sensor 1</b>		
1	Empty cavity/sensor	N/A
2	Empty sample tube (air)	N/A
3	Silver nitrate (powder)	208-1260
4	Silver oxide (powder)	180-1200
5	Silver nitrate with polymer labelled as S1D as a source of contamination (50%)	208-1260 (silver nitrate) + 300-400 (polymer)
<b>Samples tested in rectangular type microwave sensor 2</b>		
6	Empty cavity/sensor	N/A
7	Empty sample tube (air)	N/A
8	Silver nitrate (powder)	208-1260
9	Silver oxide (powder)	180-1200
10	Silver nitrate with polymer labelled as S1D as a source of contamination (50%)	208-1260 (silver nitrate) + 300-400 (polymer)

## 5. Experimental procedure and measurements

Both the S-parameters,  $S_{11}$  and  $S_{21}$ , were initially measured over the full spectrum range of the VNA between 9 KHz and 13.6 GHz frequency for all the silver samples. This was to see which of these parameters give us the best results to identify the materials and their relevant properties. For this purpose the spectrums obtained over the full range were studied further in detail to identify the resonant peaks within the narrow frequency range. All the measurements were carried out using the microwave input power of 0 dBm (1mW), the signal bandwidth set to 10 kHz and 4,000 data points over the measured frequency range (for the signal to be of high quality). The measurements were carried out in a temperature controlled room set at 20°C to obtain consistent results. Since the microwave sensor technology is robust and real-time in nature, the results for each of the measurement was obtained in 5-10 seconds.

It is also important for an industrial application that the solution has to be simple and cost effective. In this regard, the area of interest need to be identified and narrowed in terms of the frequency range (to keep the cost of the microwave source lower). In addition, the measurements taken over the full range of the spectrum has both complexities at higher frequencies and unacceptable noise levels at low frequencies. To avoid higher order modes, complexities in the measurements and the cost associated with the development of sensors



at high frequencies suitable for the industrial use, the frequency above 6 GHz was eliminated from the full spectrum obtained. Furthermore, taking into account the noise levels or response of the material to the microwave, frequency below 1 GHz was removed in the detailed analysis (section 6). The detailed material response to microwaves, resonant peaks as a result and capability of the sensors to distinctively identify the materials are shown and discussed in the results and discussion section. It can be noticed in these initial results that the sensor had a potential to identify various properties of the silver products under test, hence a proof of concept.

## 6. Results and discussion

As mentioned earlier, results of both the measurands  $S_{11}$  and  $S_{21}$  for the microwave sensor 1 and microwave sensor 2 were narrowed down to highlight the response resonant peaks of interest. The identified resonant peaks from sensor 1 for both the  $S_{11}$  and  $S_{21}$  parameters are shown in Figure 4 and 5 respectively. It should be noted that the peaks were identified as per the measurement type, i.e.  $S_{11}$  from 4.8-5.5 GHz and  $S_{21}$  from 1-6 GHz.

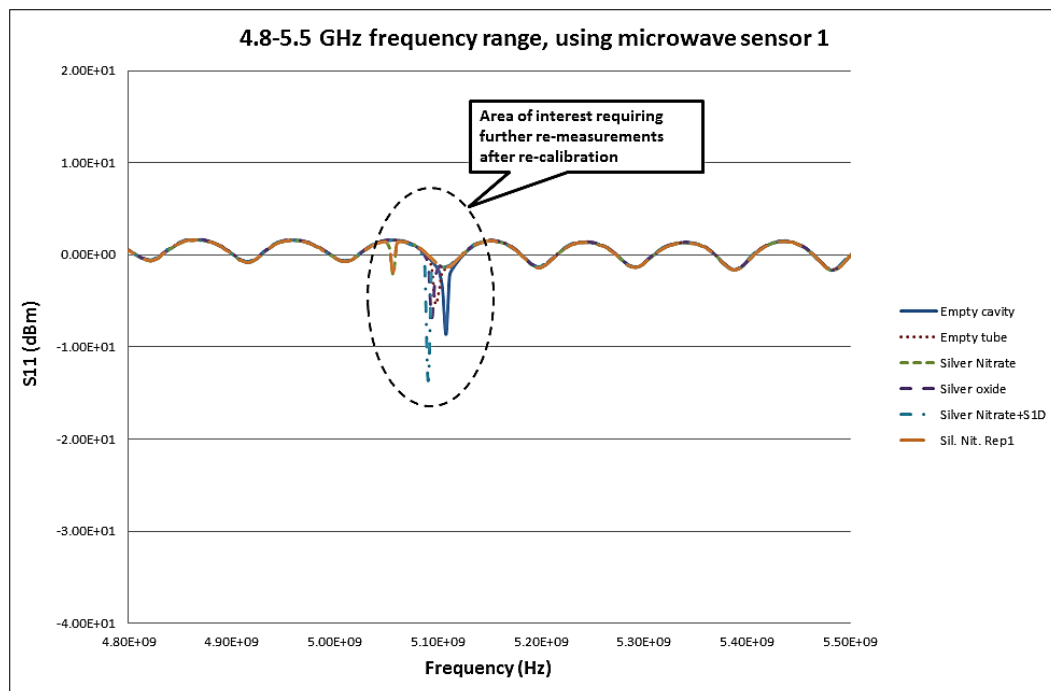


Figure 4: Reflected power,  $S_{11}$  (dBm) measurements from microwave sensor 1, 4.8-5.5 GHz

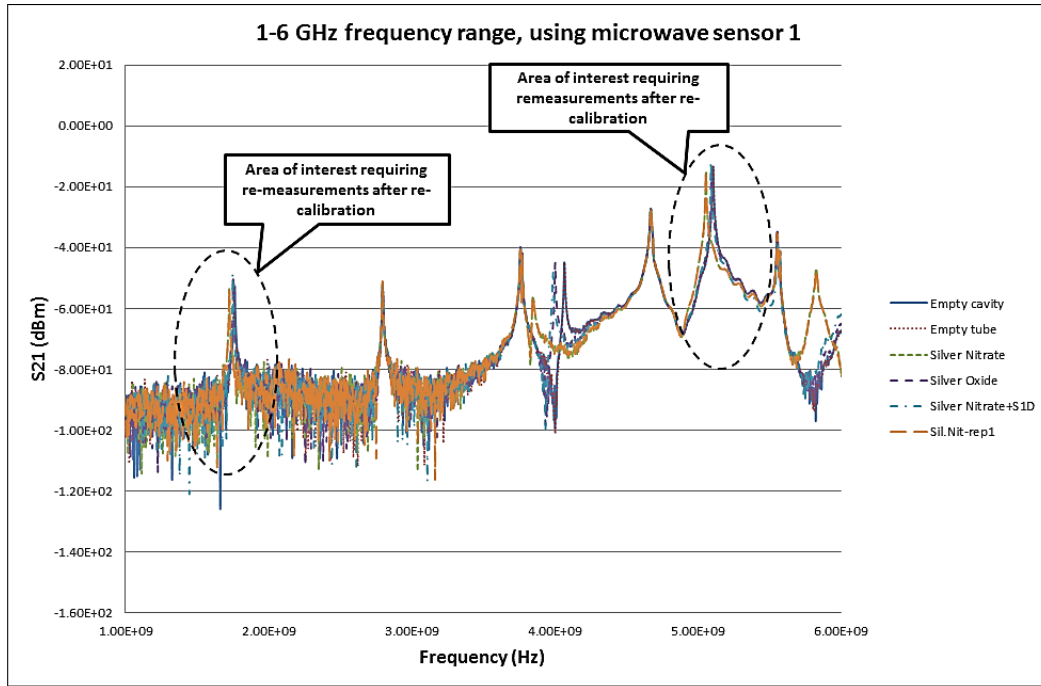


Figure 5: Transmitted power,  $S_{21}$  (dBm) measurements from microwave sensor 1, 1-6 GHz

Similarly, for microwave sensor 2, the resonant peaks identified in case of both the  $S_{11}$  and  $S_{21}$  parameters are shown in Figure 6 and 7 respectively. Once again, the frequency range for the peaks varied depending on the type of measurements, i.e.  $S_{11}$  from 2-5 GHz whereas  $S_{21}$  from 1.4-5 GHz respectively.

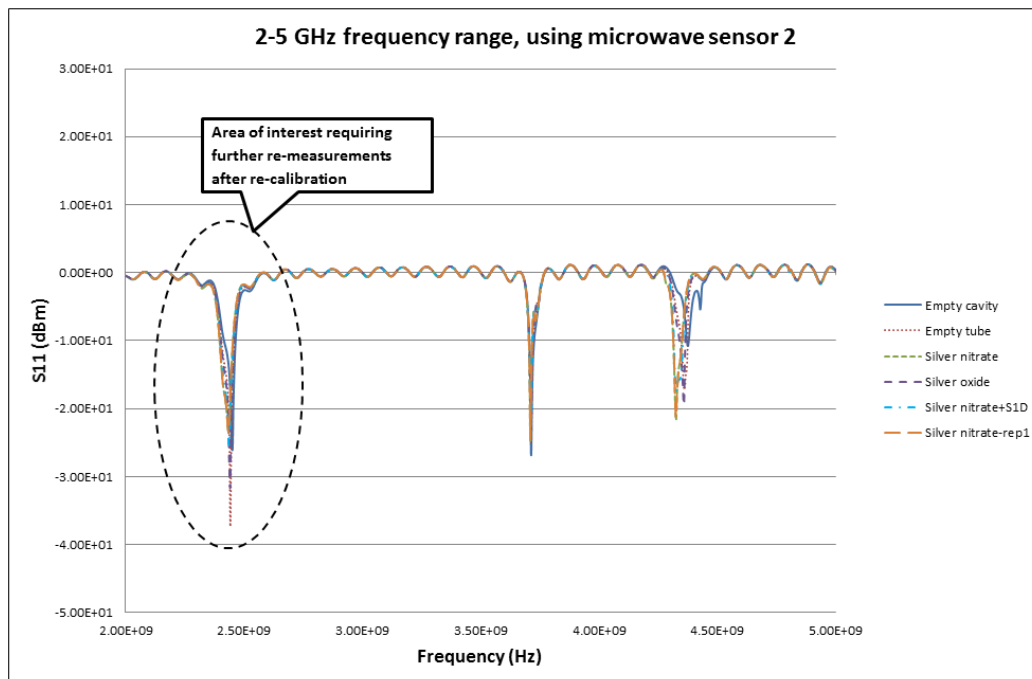


Figure 6: Reflected power,  $S_{11}$  (dBm) measurements from microwave sensor 2, 2-5 GHz

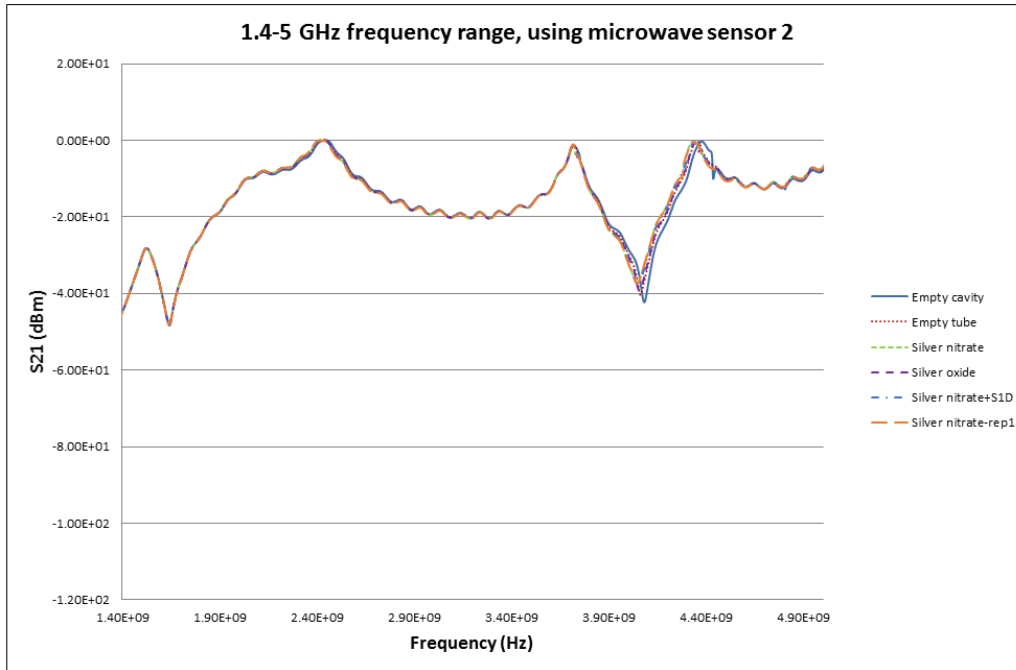


Figure 7: Transmitted power,  $S_{21}$  (dBm) measurements from microwave sensor 2, 1.4-5 GHz

It can be noticed from Figure 4-7 that the frequency range in which the resonant peaks are identified varies depending upon the type of resonant cavity sensor including its dimensions and size, as well as the areas where modes are generated inside the cavities on the application of microwave signal.

### 6.1. Re-calibration and measurements

The VNA instrument was calibrated to obtain the results from Figure 4-7. After identifying the resonant peaks, as per the highlighted areas in Figure 4-7 the instrument (VNA) was re-calibrated to study the highlighted frequency range and to achieve better microwave response to the material interaction. In the case of microwave sensor 1, the resonant peak (response of material to microwave) was detected between 5-5.15 GHz for  $S_{11}$  parameter as per in Figure 4 and presented in Figure 8.

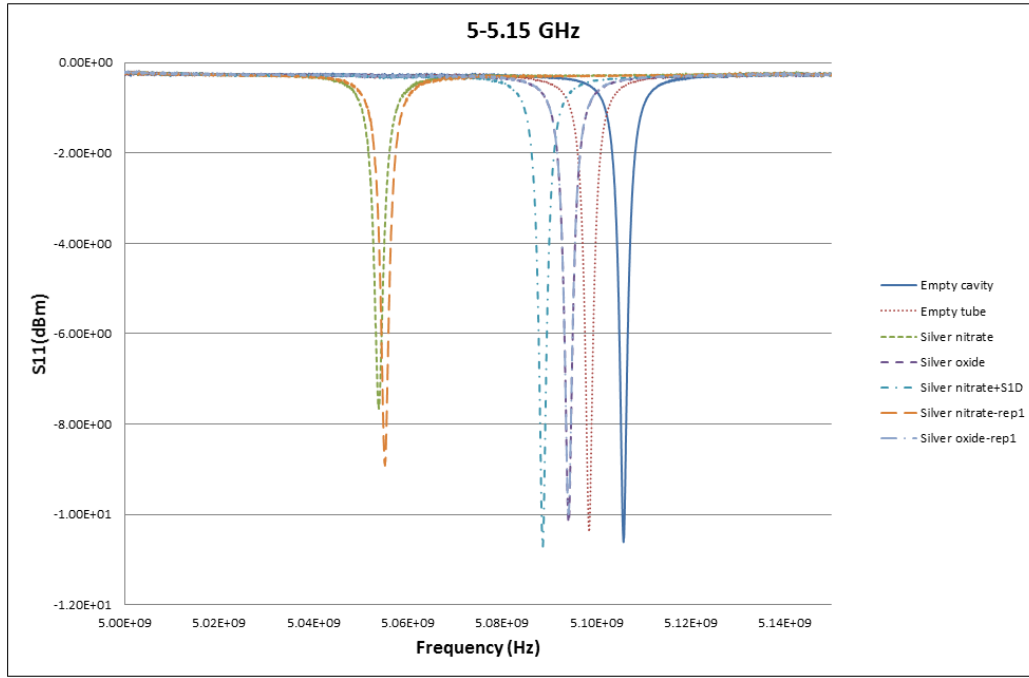


Figure 8: Reflected power,  $S_{11}$  (dBm) measurements from microwave sensor 1, 5-5.15 GHz

Figure 8 when analysed showed that each of the material has a distinctive resonant peak with a high quality factor. Empty cavity and empty tube were taken as the control samples. This was to see how much shift in the frequency and change in the amplitude occurs on introducing the silver based samples. The peaks of the control samples were at 5.1058 and 5.0985 GHz for both the empty cavity and empty tube respectively, a shift of approximately 7.3 MHz between them. This shows the sensitivity of the sensing technology to detect a material after a polypropylene sample tube was inserted into the cavity. When looking at the peak resonant frequency of silver nitrate, it was identified at 5.0540 GHz. The shift in the frequency was to the left from the empty tube and was approximately 44.5 MHz which is significant. To test the repeatability and accuracy of the microwave sensing technique immediately, the silver nitrate sample was retested resulting in the peak very close to the original signal. The slight difference could be caused probably due to the instrumentation error. This demonstrates that the technique is repeatable and can incur minimum error if the sensor is designed carefully, a detailed repeatability follows. In case of the silver oxide the resonant peak was at 5.0942 GHz. The shift of the spectrum frequency from the empty tube sample was about 4.3 MHz which is reasonably measureable keeping in mind the sensitivity of the sensor. When repeated, the resonant peak was generated at the same frequency showing promising result in terms of accuracy of the sensor and its practical application. An

additional sample was tested consisting of a blend of Silver nitrate and a polymer powder to see the feasibility of using microwave sensor to detect the contamination or foreign material. The resonant peak of the sample was detected in between Silver nitrate and Silver oxide samples. The peak was at 5.0887 GHz and the shift of the peak from the empty tube sample was to the left by a value of 9.8 MHz. The signal peak was to the right of the silver nitrate showing an addition of some foreign material to the pure silver nitrate product and was to the left from the silver oxide spectrum. This change in the spectrum could also be attributed to the change in the size/size distribution. However, this needs further investigation.

The amplitude change was also observed in addition to the frequency shifts. Both these changes in frequency and amplitude could be used to represent the difference in the material type and the existence of the foreign material (polymer) acting as a contamination source. In the case of the blend of silver nitrate and polymer, it can also be attributed to the change in the size of the sample contents. Although the results demonstrate promising output and capability of the microwave sensing technique to detect these changes, it requires further investigation through a design of a dedicated sensor to closely look at these changes and link them with the individual properties causing the changes.

On careful analysis of the  $S_{21}$  parameter representing the transmitted power of the microwave sensor 1 as in Figure 5, it was found that the material response to the microwave was more obvious at two frequency ranges, i.e. 1.66-1.8 GHz and 4.9-5.3 GHz frequency range as shown in Figure 9 and 10 respectively. A similar pattern of shifts to Figure 8 was observed for all the samples in Figure 9.

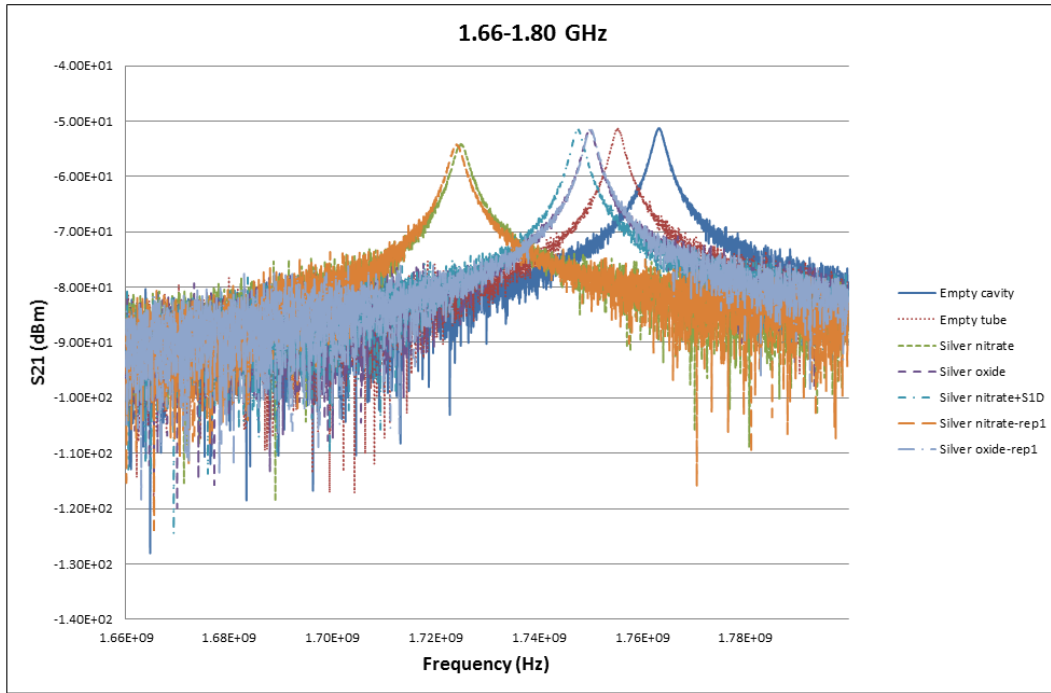


Figure 9: Transmitted power,  $S_{21}$  (dBm) measurements from microwave sensor 1, 1.66-1.8 GHz

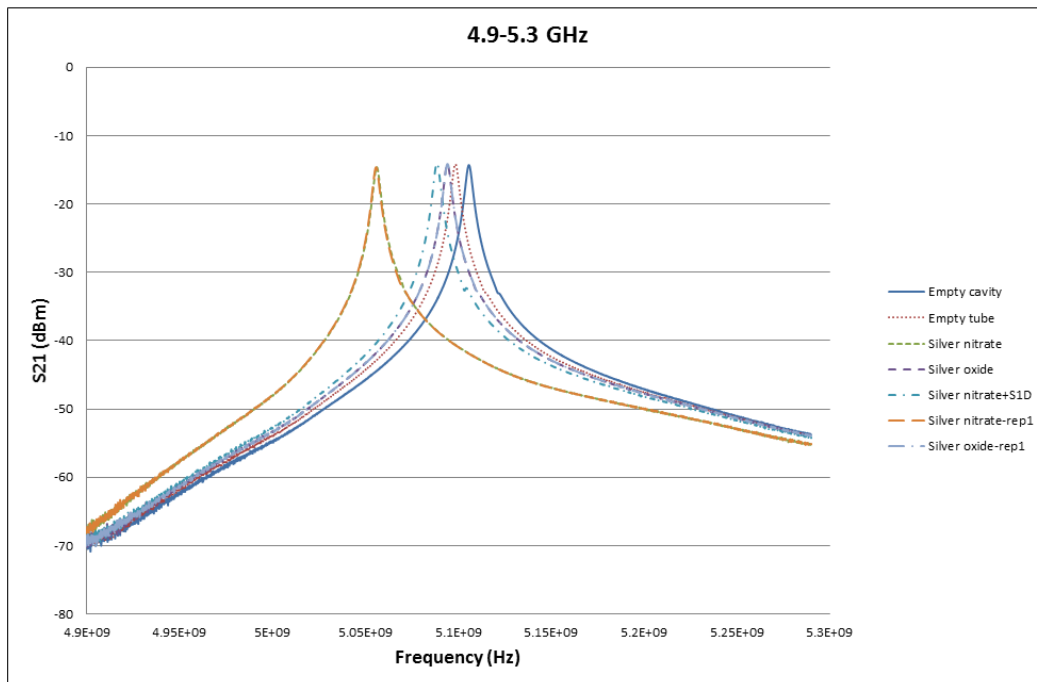


Figure 10: Transmitted power,  $S_{21}$  (dBm) measurements from microwave sensor 1, 4.9-5.3 GHz

A significant shift of the spectrum was observed in Figure 9 to the left from the control samples, i.e. empty cavity and empty tube. The maximum shift occurred in the case of silver

nitrate and silver nitrate when repeated, both almost overlapping each other. The shift was towards left similar to the results in  $S_{11}$  parameter in Figure 8. The peak frequency of the silver nitrate sample was detected at 1.6890 GHz to the left of the empty cavity at 1.6648 GHz and empty tube at 1.6654 GHz. This was a difference of approximately 24.2 MHz and 23.6 MHz respectively from the empty cavity and empty tube samples. Silver oxide peak was detected at 1.6699 GHz again to the left of the control samples. The difference was approximately 5.1 MHz from the empty cavity and 4.5 MHz from the empty tube sample. The initial repetition showed approximately similar results overlapping the original spectrums. When looking at the blend of silver nitrate and polymer, the peak was at 1.6692 GHz, 4.4 MHz to the left from the empty cavity and 3.8 MHz to the left from the empty tube sample. When analysing Figure 10 it can be observed that the resonant frequency for the empty cavity and empty tube sample was detected at approximately 5.1 GHz and 5.0974 GHz respectively. When comparing it with the silver products, the peak for the silver oxide sample was at 5.0933 GHz representing a shift of approximately 4.1 MHz to the left. On the other hand, the peak resonant frequency for the silver nitrate sample was measured at around 5.0545 GHz with a significant shift to the left of approximately 42.9 MHz from the empty tube sample. The resonant peak of the blend of silver nitrate contaminated with the polymer sample was measured and the peak was detected at approximately 5.0889 GHz. This value was between the silver nitrate and silver oxide sample whereby silver oxide was to the right of the silver nitrate and polymer blend and silver nitrate was to the left of the blend. The shift from the silver nitrate sample to the right showed the contamination.

A deep analysis was carried out to show the repeatability of the observed results. The measurements were repeated 5 times in addition to the first analysis shown in Figure 8-10. The measurements of both the  $S_{11}$  and  $S_{21}$  parameter were carried out to observe the frequency shift and the amplitude change in the case of each sample. The average of the resonant peaks for the repeated results were taken and the frequency shift calculated for the averaged results for each of the sample. Some of the results are presented in Table 2 to evident the repeatability of the results in Figure 8 & 10. It can be seen from the results that the outcome in case of the sensor 1 is very consistent and aligns with the measurements discussed above in terms of both the resonant peaks and the shifts observed with change in

the material. Negligible changes in the frequency shift after repetitions were observed between the empty tube samples and the material samples in comparison to Figure 8 & 10.

Table 2: Results of repetition of the samples indicating the resonant peaks and the frequency shifts for both the  $S_{11}$  and  $S_{21}$  parameters of the sensor 1

Sample type	Measurement type	Frequency range (GHz)	Resonant frequency (GHz)	Frequency difference/Shift (MHz) from the Empty tube sample
Empty cavity	$S_{11}$	5-5.15	5.1060	0
Empty tube			5.0975	8.5
Silver oxide			5.0934	4.1
Silver Nitrate			5.0546	43.0
Silver Nitrate + S1D			5.0883	9.2
Sample type	Measurement type	Frequency range (GHz)	Resonant frequency (GHz)	Frequency difference/Shift (MHz)
Empty cavity	$S_{21}$	4.9-5.3	5.1038	0
Empty tube			5.0979	5.9
Silver oxide			5.0925	5.4
Silver Nitrate			5.0546	43.3
Silver Nitrate + S1D			5.0891	8.8

The results from Figure 8, 9, and 10 showed pretty consistent trend in the shift of the microwave spectrums each of which indicated an individual and identifiable sample type. The measurements were consistent and repeatable as well, as in Table 2. A database can be built from the spectrums and neural network techniques can be used for further elaboration of the results when designing a bespoke sensor.

The results of the  $S_{11}$  parameter from the rectangular microwave sensor 2 were also promising, resulting in the sharp resonant peaks for the individual samples. From the results of Figure 6 the frequency range of 2.26-2.61 was identified and highlighted as a point of further investigation. The instrument was re-calibrated and measurements re-taken around this frequency. The results are shown in Figure 11.

On careful study of the result in Figure 11, it can be seen that each of the material has a distinctive resonant peak with a high quality factor. The peaks of the empty cavity and empty tube were at 2.4416 and 2.4321 GHz respectively. When looking at the peak resonant



frequency of the silver oxide identified at 2.4294 GHz, the shift of the frequency from the empty tube was approximately 2.7 MHz which is considerable again keeping in mind the sensitivity of the measurement technique. The Silver oxide sample was immediately retested resulting in the peak occurring exactly at the same frequency as the original to show the instant repeatability of the results. This demonstrates that the technique is repeatable (a detailed repeatability follows) and can incur minimum error despite the sensor type. In the case of silver nitrate sample the peak frequency was identified at 2.4224 GHz. The shift of the frequency from the silver oxide sample was approximately 7 MHz again resulting in a measurable shift. When repeated, the resonant peak was generated at the same frequency. A contaminated sample was also tested with this sensor consisting of silver nitrate and a polymer powder.

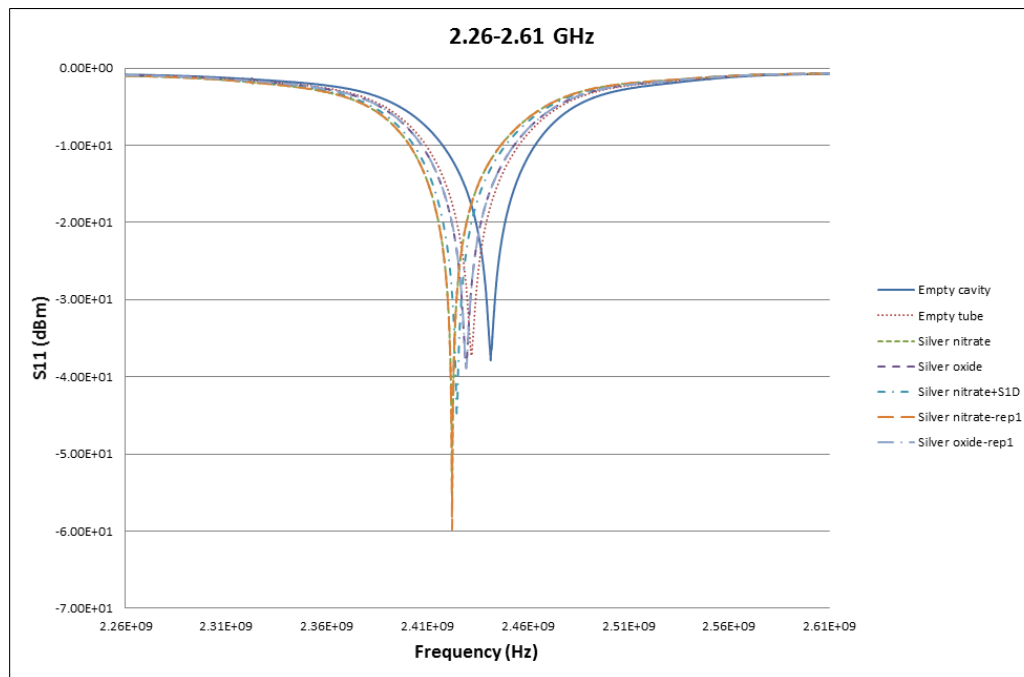


Figure 11: Reflected power,  $S_{11}$  (dBm) measurements from microwave sensor 2, 2.26-2.61 GHz

The resonant peak was detected in between the silver nitrate and the silver oxide samples. The shift of the peak was towards the right from the silver nitrate showing an addition of some foreign material to the pure silver nitrate. The peak was detected at 2.4247 GHz, a 2.3 MHz shift to the right of the silver nitrate, and sufficient to differentiate between the samples due to the sensitivity of the technique. To further detail the repeatability and accuracy of the

sensing technique, the samples were repeated 5 times in addition to the measurements shown in Figure 11 and the results presented in Table 3. The resonant peaks for each of the material type were averaged and the shifts calculated. It can be observed that the results were showing similar trend as in Figure 11 with reliable and repeatable resonant peaks as well as the associated frequency shifts.

Table 3: Results of repetition of the samples indicating the resonant peaks and the frequency shifts for the  $S_{11}$  parameter of the sensor 2

Sample type	Measurement type	Frequency range (GHz)	Resonant frequency (GHz)	Frequency difference/Shift (MHz) from the Empty tube sample
Empty cavity	$S_{11}$	2.26-2.61	2.4487	0
Empty tube			2.4412	7.5
Silver oxide			2.4393	1.9
Silver Nitrate			2.4319	9.3
Silver Nitrate + S1D			2.4342	7.0

In the case of  $S_{11}$  measurements, amplitude change was also identified in addition to the frequency shifts in the case of each sample. As in Figure 8, these changes in the frequency and amplitude can be attributed to the difference in the material type, the existence of the foreign material (polymer) acting as a contamination source and to the change in the size of the sample contents as in the case of a blend of silver nitrate and polymer. Although, the results and their repetitions demonstrate promising output and capability of the microwave sensing technique these changes require further investigation to closely look at these changes and link them with the individual properties causing them.

As far as the parameter  $S_{21}$  is concerned from microwave sensor 2 (Figure 7), the results didn't provide sufficient information in terms of generating quality resonant peaks. Hence,  $S_{21}$  was not further analysed.

## 6.2. Statistical analysis

To monitor and assess the variation in the repeat measurement of each sample, standard deviation test was used for both the cavities for the results presented in the section 6.1. Each of the sample was tested for the variation in the resonant peak obtained on repetition. The results were promising and the variation in the standard deviation obtained from the 5 times

repeated measurements was less than 0.001% from the corresponding averaged resonant frequency of the sample. This shows the accuracy of the sensor when the measurement was repeated. The variation can be improved further by a careful design of a bespoke cavity sensor unit. The results are presented in Figure 12 for the two sensors along with the percentage of deviation from the average value in Table 4.

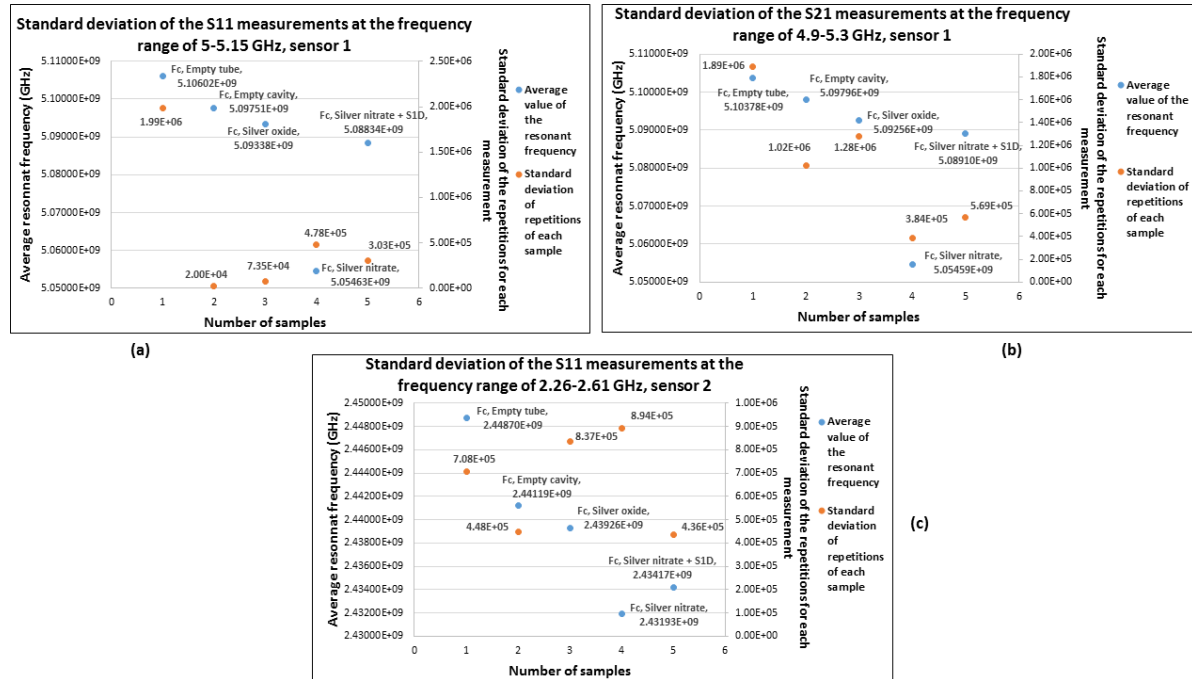


Figure 12: Averaged resonant frequency and the corresponding standard deviation of the 5 times repeated measurements of each sample (a)  $S_{11}$  measurements of the sensor 1, frequency: 5-5.15 GHz (b)  $S_{21}$  measurements of the sensor 1, frequency: 4.9-5.3 GHz (c)  $S_{11}$  measurements of the sensor 2, frequency: 2.26-2.61 GHz

Table 4: Averaged resonant frequency and the Standard deviation test results of the accuracy of repetitions along with the percentage in the deviation from the averaged values

Sample type	Measurement type (sensor 1)	Frequency range (GHz)	Averaged resonant frequency (GHz)	Standard deviation $\sigma = \sqrt{\frac{\sum(x-\mu)^2}{N}}$ (MHz)	Deviation in % from the averaged resonant frequency
Empty cavity	S <sub>11</sub>	5-5.15	5.1060	1.99	<0.0003%
Empty tube			5.0975	0.02	<0.00001%
Silver oxide			5.0934	0.0735	<0.00001%
Silver Nitrate			5.0546	0.478	<0.0001%
Silver Nitrate + S1D			5.0883	0.303	<0.0001%

Empty cavity	S <sub>21</sub>	4.9-5.3	5.1038	1.89	<0.0004%
Empty tube			5.0979	1.02	<0.0003%
Silver oxide			5.0925	1.28	<0.0003%
Silver Nitrate			5.0546	0.384	<0.00008%
Silver Nitrate + S1D			5.0891	0.569	<0.0001%
Sample type	Measurement type (sensor 2)	Frequency range (GHz)	Averaged resonant frequency (GHz)	Standard deviation (MHz) $\sigma = \sqrt{\frac{\sum(x-\mu)^2}{N}}$	Deviation in % from the averaged resonant frequency
Empty cavity	S <sub>11</sub>	2.26-2.61	2.4487	0.708	<0.0002%
Empty tube			2.4412	0.448	<0.0001%
Silver oxide			2.4393	0.837	<0.0004%
Silver Nitrate			2.4319	0.894	<0.0004%
Silver Nitrate + S1D			2.4342	0.436	<0.0001%

## 7. Conclusions and Recommendations

An initial feasibility study was carried out to assess the capability of using a novel, robust and an instantaneous microwave sensing technique to analyse silver based products. The results were promising exhibiting accuracy and repeatability that are the attributes required for any industrial application. To extensively test the repeatability and reliability of the microwave sensing technique, a detailed repetition of the samples was carried out to monitor the resonant peaks generated and the frequency shifts. The results were very consistent with the initial graphs presented in Figure 8-11. Results of some of the repetitions i.e. Figure 8, 10 and 11 are also presented in Table 2 and 3. It is clear from the results that the technique is capable of detecting various properties of silver products such as their type, contamination, particle size and/or particle size distribution, etc. Further research work, however, is required to design and develop a dedicated unit with minimum errors and to further explore the capability of detecting multiple parameters, linking them back to the material itself. Potentially, the technique can be further developed as an alternative to conventional time consuming physical and chemical testing methods.

Analysis of individual peaks and smaller sections enabled the investigation to find the capability of microwave sensors to differentiate between samples. The following are some of the conclusions and recommendations from the investigation.

- The sensor spotted the individual samples of silver oxide and silver nitrate showing the difference between the samples and contamination in them along with the size change in the blend (as in Table 1).
- The differences between the samples were noticeable, however, these results although promising and showing the potential of microwave sensing to do the silver products analysis, yet need further investigation through the design of a dedicated sensor that can eliminate any errors encountered in using the microwave sensor in this study. The recommended further investigation will help to relate the changes due to material interaction with microwaves to specific properties of silver based materials.
- An in-depth repeatability of the measurements showed that the output was reliable and the results consistent with the initial measurements. The results presented in Table 2 and 3 showed both the resonant peaks and the frequency shift patterns to evidence the claim.
- The results of the standard deviation test (Figure 12 and Table 4) showed a high accuracy of both the sensors in the case of repetition. The percentage of deviation from the averaged resonant frequency was very small. This can be improved further by a careful design of the bespoke sensor unit.
- Further investigation will also assist in building up a database containing all the microwave spectrums captured from the measurements for the future reference and analysis. It will also help in targeting the anomalies in the existing technique and sensor itself to provide more accurate results. The results stored can then be related to various properties of silver based materials.

From the results and discussion it has been demonstrated that microwave sensing can be developed as a technology to assist the industry dealing with silver based products. The results propose a next stage of the study whereby a bespoke prototype sensor with appropriate dimensions is suggested to be designed and developed with the aim to target the industrial needs. This could have a positive and significant impact on the quality control and assurance process at Amesgoldsmith Ltd in specific. In general, the prototype will provide a potential option to the industry dealing with silver based products in terms of analysing and testing batch of samples instantaneously under operational time constraints. Microwave

sensing technique will also be helpful in assessing the quality and validate the products within the tight constraints that exist to assure the quality of the product. This can be done with minimum costs and more accurately through this technique. The cost effectiveness comes from designing a unit operating within a lower narrow band of frequency range operating at a very low power. The project can be carried out in collaboration and can be phased to target priority requirements of the industrial partner.

## **Acknowledgements**

The authors would like to acknowledge European Regional Development Fund (ERDF) for funding the Low Carbon Innovation Hub project. The authors also acknowledge Liverpool John Moores University's Radio Frequency & Microwave (RFM) Group for providing their designed cavity resonator for the feasibility study. The authors would like to say thanks to AmesGoldsmith UK Ltd for their collaborative efforts and continuous support in various aspects of the study including providing material samples and relevant information for testing. Our Thanks would also go to our colleagues who supported us in any form to carry out this feasibility. This work was supported by European Regional Development fund (ERDF) [X03166PR]

## **Disclosure statement**

It is acknowledged that there is no direct financial interest arising from this feasibility study. However, development of a dedicated sensor may benefit to the industry dealing in the characterisation of Silver products both in terms of the cost and time savings.

## **8. References**

- [1] J.H. Goh, A. Mason, A.I. Al-Shamma'a, Non-Invasive microwave sensor for the detection of Lactic acid in Cerebrospinal fluid (CSF), Journal of Physics: Conference Series 307 01 (2011) 6pp.
- [2] O. Korostynska, A. Mason, A. I. Al-Shamma'a, Flexible microwave sensors for real-time analysis of water contaminants, Journal of Electromagnetic Waves and Applications 27 16 (2013) 2075-2089.
- [3] D.J. Rowe, J. Naylor, A. Porch, D.A. Barrow, C.J. Allender, Microwave resonant sensor for real-time continuous-flow measurements of microfluidic systems, 14<sup>th</sup> International

Conference on Miniaturized Systems for Chemistry and Life Sciences, 3-7 October 2010, Groningen, The Netherlands.

[4] K.Y. You, J. Salleh, Z. Abbas, L.L. You, A rectangular antenna technique for the determination of moisture content in soil, Progress in Electromagnetics Research Symposium Proceedings (PIERS), 5-8 July 2010, Cambridge, USA.

[5] R. Wellock, A.D. Walmsley, Applications of microwave spectroscopy in process analysis, Process Column, Clairet Scientific: Northampton, UK (2004),  
[www.clairet.co.uk/downloads/PSC09.pdf](http://www.clairet.co.uk/downloads/PSC09.pdf) <Last accessed: 25/09/2015>.

[6] Mason, A., Korostynska, O., Ortoneda-Pedrola, M., Shaw, A., and Al-Shamma'a, A. A resonant co-planar sensor at microwave frequencies for biomedical applications Sensors and Actuators A: Physical 202 (2013) 170-175.

[7] Agilent, Microwave dielectric spectroscopy workshop: Measure the difference, Copyright © Agilent technologies Inc., USA, (2004) 1-42.

[8] M.A. Al-Kizwini, S.R. Wylie, D.A. Al-Khafaji, A.I. Al-Shamma'a, The monitoring of the two phase flow-annular flow type regime using microwave sensor technique Measurement 46 (2013) 45-51.

[9] Blakey, R., Korostynska, O., Mason, A., and Al-Shamma'a, A Real-time microwave based sensing method for vegetable oil type verification Procedia Engineering 47 (2012) 623-626.

[10] A. bababjanyan, H. Melikyan, S. Kim, J. Kim, K. Lee, B. Friedman, "Real-time noninvasive measurement of glucose concentration using a microwave biosensor Journal of Sensors 2010 (2010) 7 pages.

[11] S.R. Wylie, A. Shaw, A.I. Al-Shamma'a, RF sensor for multiphase flow measurement through an oil pipeline Measurement Science and Technology 17(2006) 2141-2149.

[12] M. Ateeq, A. Senouci, H. Al-Nageim, A. Al-Shamma'a, Microwave spectroscopy for the analysis of absorption properties of treated waste rubber aggregates Journal of Hazardous, toxic, and radioactive waste 16(4), 2012, 334-342.

[13] M. Ateeq, s. Wylie, A. al-Shamma'a. H. Al-Nageim, Microwave spectroscopy: a potential technique to analyse bitumen dielectric and physical properties Measurement Science and Technology 23(2012) 13pp.

[14] M. Muradov, J.D. Cullen, B. Abdullah, M. Ateeq, A. Mason, A. Shaw, A. Al-Shamma'a, Real-time monitoring of meat drying process using microwave spectroscopy Proceedings of the 8th International Conference on Sensing Technology, 2-4 September 2014, Liverpool, UK.